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APPLICATION FOR LETTERS PATENT

for

GELLING AGENTS OR THICKENERS

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TITLE OF THE INVENTION
GELLING AGENTS OR THICKENERS

CROSS-REFERENCE TO RELATED APPLICATION

[0001] This application is a continuation of PCT International Patent Application PCT/NL02/00151, filed March 6, 2002, designating the United States and published, in English, as International Publication Number WO 02/070463 A1, filed September 12, 2002, the contents of which are incorporated by this reference.

TECHNICAL FIELD

[0002] The present invention relates to a novel class of gelling agents, a process for producing them and to their application in preparing gels for various applications.

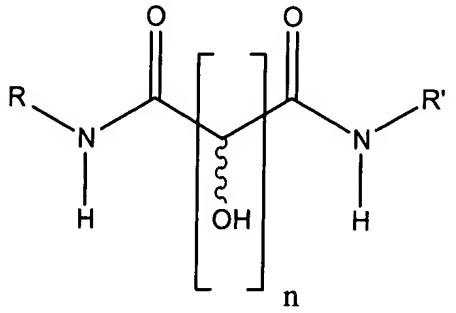
BACKGROUND

[0003] Thermally reversible gelling or thickening of organic solvents by low molecular weight compounds are of particular interest for hardeners of spilled fluids and cooking oils, thickeners for paints, cosmetic materials and several other technical applications. The self assembly of these gelator/thickener molecules occurs by means of noncovalent interactions such as hydrophobic interaction, π-π interactions, electronic interactions, hydrogen bonding or combinations thereof. Although several gelator/thickener molecules have been identified during the last decade, there is still interest in stable gelator/thickeners that can be synthesized easily from cheap, renewable sources and gelate or thicken a wide variety of solvents.

BRIEF SUMMARY OF THE INVENTION

[0004] The present invention includes a novel class of gelling agents or thickeners. The present invention provides gelling agents or thickeners that are based on readily available and economically attractive starting materials. The present invention also provides gelling agents or thickeners capable of gelling or thickening a wide variety of solvents, making the gelling agents or thickeners suitable for employment in various applications.

[0005] The invention includes preparing gelling agents or thickeners from low molecular carbohydrates. The present invention relates to a gelling agent in the form of N,N′-disubstituted aldaramides and N,N′-disubstituted pentaramides and derivatives thereof. Specifically, the present invention relates to a gelling agent having the following structure:



(I),

wherein n is 3 or 4, and wherein R and R′ represent the same or different substituents chosen from the group of substituted or unsubstituted, branched, possibly aromatic, groups containing cyclic or linear alkyl, alkenyl, alkynyl groups having from 1 to 40 carbon atoms.

BEST MODE OF THE INVENTION

[0006] In a preferred embodiment, R and R′ each represent, independently, a linear, branched, or cyclic alkyl group having 4-20 carbon atoms. More preferred is that R and R′ each are independently selected from the group of cycloalkyl groups having 4-16 carbon atoms. In a preferred embodiment, R and R′ represent the same substituent.

[0007] One advantage of the present invention is that gelling agents or thickeners can be based on naturally occurring products, such as carbohydrates. Thus, the starting materials for producing them are from a renewable source.

[0008] A gelling agent or thickener according to the present invention may be prepared by converting an aldose or pentose to its corresponding aldaric or pentaric acid, or a salt thereof, such as an alkali metal salt or an (alkyl)ammonium salt. It is preferred to use an aldose or pentose chosen from the group of allose, altrose, glucose, mannose, gulose, idose, galactose, talose, ribose, arabinose, xylose, lyxose and derivatives thereof, as these lead to products having particularly favorable gelling and/or thickening properties. It is to be noted that both the L and the D isomers of the aldose or pentose, as well as mixtures thereof, can be used. Suitable

derivatives of the mentioned aldoses and pentoses include deoxy aldoses or pentoses, ethers, esters and the like. In a more preferred embodiment, D-glucose is chosen as aldose.

[0009] The conversion of the aldose or pentose to its corresponding aldaric or pentaric acid is generally achieved by oxidation. The oxidation can suitably be carried out using Pt/O_2, TEMPO/NaOCl/(NaBr) or $HNO_3/(NaNO_2)$ as an oxidizing agent. Further details for the manner in which the oxidation may be carried out can be found in U.S. Patents 5,831,043, 5,599,977 and 6,049,004, and in *J. Org. Chem.*, 1977, 42, 3562-3567; J-F. Thaburet *et al.*, *Carbohydr. Res.* 330 (2001), 21-29, all of which are incorporated herein by reference.

[0010] The thus obtained aldaric or pentaric acid or salt thereof is subsequently condensed with a primary amine to obtain the objective gelling agent or thickener.

[0011] The aldaric or pentaric acid can be condensed with an amount of at least 200 mole%, with respect to the aldaric or pentaric acid, of a primary amine. It is preferred to activate the aldaric or pentaric acid first by means of lactonization and/or esterification, depending on the stereochemistry of the carbohydrate. Further details may be found in Kurtz *et al.*, *J. Biol. Chem.*, 1939, 693-699; Hoagland, *Carbohydrate Res.*, 1981, 98, 203-208, and United States Patent 5,312,967, which are incorporated herein by reference.

[0012] In an alternative embodiment, nonsymmetrical N,N′-dialkylaldaramides or N,N′-dialkylpentaramides may be prepared, wherein R and R′ represent different substituents. In accordance with this embodiment, the aldaric or pentaric acid may be converted into an N-alkyl-1-aldar/pentaramid-6-ate or N-alkyl-6-aldar/pentaramid-1-ate (as disclosed in U.S. Patent 5,239,044; L. Chen *et al.*, *J. Org. Chem.*, 61 (1996) 5847-5851; R. Lee *et al.*, *Carbohydr. Res.* 64 (1978) 302-308; and K. Hashimoto *et al.*, *J. Polym. Sci. Part A, Polym. Chem.*, 37 (1999) 303-312), activated and subsequently condensed with, preferably 100 mole% with respect to the N-alkyl aldar/pentar-ate, of a second primary amine.

[0013] In general, the obtained gelling agent or thickener precipitates from the reaction mixture in which it is formed and can be easily isolated by filtration. Further purification can be performed by conventional techniques like crystallization or, in the case of products based on galactaric acid derivatives, by thoroughly washing with ethanol, water, acetone or hexane.

[0014] It will be understood that the use of the present gelling agents or thickeners to prepare a gel or to thicken a composition is also encompassed by the invention. As is well

known, gelling behavior of compounds or compositions is highly unpredictable. In principle, a solution of a specific compound in a solvent, e.g. an organic solvent, is considered a gel when a homogeneous substance is obtained which exhibits essentially no gravitational flow. Preferably, the gelling phenomenon is thermo reversible. However, in so far as the present compounds do not provide a gel in a composition, they may be used as a thickener or rheology controlling agent, as their addition to a composition may give rise to an increase in viscosity of the composition.

[0015] Compositions in which the present compound has been found to produce a gel include compositions in numerous solvents. Preferred examples include aromatic and nonaromatic hydrocarbons, alcohols, ethers, esters, aldehydes, alkanoic acids, epoxides, amines, halogenated hydrocarbons, silicon oils, vegetable oils, phosphoric esters, sulfoxides, water and mixtures thereof. In order to obtain a gel, the gelling agent or thickener is preferably mixed with the composition to be transformed to a gel in an amount of between 0.01 and 50 wt.%, based on the weight of the composition. In a preferred embodiment, the mixture of the gelling agent or thickener and the composition is heated to allow for an even better gel formation or thickening. Typically, the heating will involve raising the temperature of the mixture to about 30 - 175° C until a clear solution is obtained. In an alternative embodiment, the gelling agent is first dissolved in a polar or apolar solvent and then added to or sprayed into a composition or solvent to be converted into a gel.

[0016] The resultant gel or thickened composition, which is also encompassed by the present invention, may find use in one of numerous applications. It is conceived that such applications lie in the field of cosmetics, oil recovery (*e.g.* from the sea), food products, transport of industrial solvents, stabilization of organic solvents under near zero gravity conditions, stiffening of fuels to increase stability and reduce fluidity, lubricants, coatings, printing inks, and adhesives. In these applications, they may be used analogous to conventional gelling agents or thickeners, which they replace.

[0017] The invention will now be further elucidated by the following, illustrative examples.

EXAMPLES

Synthesis of starting materials

[0018] Potassium hydrogen D-glucarate (R.L. Whistler, M.L. Wolfrom, J.N. BeMiller, *Methods in Carbohydrate Chemistry*, Vol. II (1963), Academic Press Inc., 47-48), D-glucaric acid (lactone) (L. Chen, D.E. Kiely, *J. Org. Chem.*, 61 (1996) 5847-5851), D-glucaro-6,3-lactone (L. Chen, D.E. Kiely, *J. Org. Chem.*, 61 (1996) 5847-5851), D-mannaric acid dilactone (E. Fischer, Berichte, 24 (1891) 539-546), diethyl galacterate (R.L. Whistler, M.L. Wolfrom, J.N. BeMiller, *Methods in Carbohydrate Chemistry*, Vol. II (1963), Academic Press Inc., 40-41), D-ribaric acid (lacton) (C.E. Cantrell, D.E. Kiely, G.J. Abruscato, J.M. Riordan, *J. Org. Chem.*, 42 (1977) 3562-3567, as described for D-xylaric acid, R.E. Gall, L. Tarasoff, *Aust. J. Chem.*, 28 (1975) 687-691) were synthesized according to literature procedures.

Cyclohexylammonium 6-(N-cyclohexyl)-D-glucaramide-1-ate.

[0019] D-glucaro 6, 3-lacton (1.04 g, 5.4 mmol) was added to a solution of cyclohexylamine (1.34 g, 13.5 mmol) in EtOH (50 ml). After 20 hours stirring, the precipitate was filtered off and crystallized from EtOH. Yield 0.97 g (2.5 mmol, 46%). ^{1}H-NMR (d_6-DMSO, 300 MHz, ppm, δ 1.10-1.38 (m, 10 H), 1.45-1.90 (m, 10H), 2.91 (m, 1H), 3.55 (m, 1H), 3.65 (m, 2H), 3.78 (t, 1H), 3.89 (d, 1H), 7.55 (d, 1H). ^{13}C-NMR (d6-DMSO, 300 MHz, ppm): 24.54, 25.23, 25.32, 25.85, 31.28, 32.86, 32.93, 47.88, 49.82, 71.60, 72.45, 72.80, 73.27, 172.70, 176.36. Anal Calculated for $C_{18}H_{34}N_2O_7$: C, 55.37; H, 8.78, N, 7.17. Found: C, 55.22; H, 8.76; N, 7.37.

Synthesis of 3-O-methyl diethyl D-glucaric acid.

[0020] 3-O-Methyl-∀,∃-D-glucose (7.00 g, 36 mmol) was added in portions (in 45 minutes) to a solution of $NaNO_2$ (0.010 g, 0.14 mmol) in HNO_3 (15 ml, 65%) at T = 50-55° C. After 45 minutes T = 50° C, the reaction was cooled to RT, and stirred for another 30 minutes. EtOH (40 ml) was added in portions and the reaction mixture was stripped with EtOH several times using a rotavap. The crude reaction mixture was distilled (Kugelrohr) and the fraction of b.p. 225° C/0.4 mm Hg was collected. Yield 4.74 g (16.9 mmol, 47%). ^{1}H-NMR (d_6-DMSO, 300 MHz, ppm, δ 1.20 (t, 6H), 3.31 (s, 3H), 4.10 (m, 4H + 1H), 4.35 (m, 1H), 4.49 (m, 1H), 4.94

(t, 1H). ^{13}C-NMR (d_6-DMSO, 300 MHz, ppm): 15.09, 59.50, 61.47, 69.96, 71.81, 78.06, 83.89, 171.47, 176.02.

Synthesis of citronellylamine.

[0021] 1) Preparation of 3, 7-dimethyl-oct-6-enal oxim. Citronellal (15.37 g, 100 mmol) in EtOH (300 ml) was added to a solution of NH_2OH (7.00 g, 100 mmol) and NaOH (4.07 g, 102 mmol) in H_2O (100 ml) and stirred for 20 hours at T = 60° C. After evaporation, the remaining oil was dissolved in H_2O, acidified with 2M HCl, and subsequently extracted with Et_2O (2 x). After drying with Na_2SO_4 filtration and evaporation, crude 3, 7-dimethyl-oct-6-enal oxim (mixture of cis/trans) was isolated. Yield 14.89 g (88 mmol, 88%). ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm, δ 0.88 (t, 3H), 1.11-1.34 (m, 2H), 1.54 (s, 3H), 1.62 (s, 3H), 1.94-2.30 (m, 5H), 5.01 (t, 1H), 6.69 (t, 0.5 H), 7.36 (0.5 H). ^{13}C-NMR ($CDCl_3$, 300 MHz, ppm): 15.12, 16.92, 17.21, 22.89, 22.95, 23.18, 27.98, 28.42, 29.47, 33.88, 34.11, 34.32, 121.79, 121.84, 128.96, 148.91.

[0022] 2) Preparation of citronellyl amine. 3, 7-Dimethyl-oct-6-enal oxim (mixture of cis/trans 1:1, 14.54 g, 68 mmol) was added slowly to 173 ml of a solution of 1M $LiAlH_4$ in THF under N_2 atmosphere. After 20 hours refluxing, the suspension was decanted and the precipitate was washed with Et_2O (3x). After drying of the Et_2O/THF layer with Na_2SO_4, filtration and evaporation of the solvent, the remaining oil was distilled under reduced pressure (0.8-1.0 mm Hg, T = 65° C). Yield 5.00 g (32.4 mmol, 37%). ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm, δ 0.88 (d, 3H), 0.92-1.44 (m, 7H), 1.50 (s, 3H), 1.58 (s, 3H), 1.88 (m, 2H), 2.62 (m, 2H), 5.00 (t, 1H). ^{13}C-NMR ($CDCl_3$, 300 MHz, ppm): 15.07, 17.00, 22.94, 23.15, 27.57, 34.67, 37.57, 38.68, 122.27, 128.57.

Synthesis of 8-amino-pentadecane.

[0023] 1) Preparation of pentadecan-8-one oxim. Dihexylketone (13.47 g, 68 mmol) in EtOH (300 ml) is added to a solution of NH_2OH (4.74 g, 68 mmol) and NaOH (2.74 g, 69 mmol) in H_2O (100 ml) and stirred for 20 hours at T = 60° C. After evaporation, the remaining oil is dissolved in H_2O, acidified with 2M HCl, and subsequently extracted with Et_2O (2 x). After drying with Na_2SO_4 filtration and evaporation, crude pentadecan-8-one oxim (mixture of cis/trans 1:1) was isolated. Yield 13.57 g (64 mmol, 93%). ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm, δ

0.83 (t, 6H), 1.25 (m, 8H), 1.45 (m, 4H), 2.11 (t, 2H), 2.28 (t, 2H). ^{13}C-NMR ($CDCl_3$, 300 MHz, ppm): 11.56, 20.06, 23.13, 23.76, 25.99, 26.51, 27.07, 29.09, 31.61, 159.57.

[0024] 2) Preparation of 8-amino-pentadecane. Pentadecan-8-one oxim (mixture of cis/trans 1:1, 13.45 g, 63 mmol) was added slowly to 127 ml of a solution of 1M $LiAlH_4$ in THF under N_2 atmosphere. After 20 hours refluxing, the suspension was decanted and the precipitate was washed with Et_2O (3x). After drying of the Et_2O/THF layer with Na_2SO_4, filtration and evaporation of the solvent, the remaining oil was distilled under reduced pressure (0.8-1.0 mm Hg, T = 105° C). Yield 5.17 g (26.1 mmol, 41%). ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm, δ 0.79 (d, 6H), 1.18-1.30 (m, 20H), 2.58 (m, 1H). ^{13}C-NMR ($CDCl_3$, 300 MHz, ppm): 11.53, 20.09, 23.62, 26.97, 29.35, 35.67, 48.67.

Example 1

[0025] Synthesis of dibutyl D-glucaramicle. D-Glucaric acid (lactone) (1.43 g, about 7.1 mmol) was added to a solution of butylamine (1.34 g, 17.9 mmol) in EtOH (30 ml). After 20 hours stirring, the precipitate was filtered off and crystallized from EtOH (yield 0.31 g, 1.0 mmol, 14%). ^{1}H-NMR (d_6-DMSO, 300 MHz, ppm): δ 0.87 (t, 6H), 1.28 (m, 4H), 1.40 (m, 4H), 3.08 (m, 4H), 3.69 (bs, 1H, H_4), 3.88 (bs, 1H, H_3), 3.92 (bs, 1H, H_5), 3.98 (bs, 1H, H_2), 4.61 (d, 1H, OH_3), 4.74 (d, 1H, OH_4), 5.35 (d, 1H, OH_2), 5.52 (d, 1H, OH_5), 7.59 (t, 1H, NH_1), 7.84 (t, 1H, NH_6). ^{13}C-NMR (d6-DMSO, 300 MHz, ppm): 14.65, 20.45, 32.21, 38.83, 71.33, 72.50, 73.95, 74.21, 173.01, 173.98, Anal Calculated for $C_{14}H_{28}N_2O_6$: C, 52.48; H, 8.81, N, 8.74. Found: C, 52.06; H, 8.79; N, 8.61.

Example 2

[0026] Synthesis of dibutyl D-mannaramide. D-Mannaric acid dilactone (0.61 g, 3.5 mmol) is added to a solution of butylamine (0.81 g, 10.8 mmol) in EtOH (20 ml). After 20 hours stirring, the precipitate was filtered off and crystallized from EtOH (yield 0.38 g, 1.2 mmol, 34%). ^{1}H-NMR (d_6-DMSO, 300 MHz, ppm): δ 0.87 (t, 6H), 1.24 (m, 4H), 1.39 (m, 4H), 3.09 (q, 4H), 3.70 (t, 2H, H_3, H_4), 3.88 (t, 2H, H_2, H_5), 4.79 (d, 2H, OH_3, OH_4), 5.42 (d, 2H, OH_2, OH_5), 7.84 (t, 2H, NH_1, NH_6). ^{13}C-NMR (d_6-DMSO, 300 MHz, ppm): 14.64, 20.45, 32.13,

38.59, 72.09, 72.37, 174.36. Anal Calculated for $C_{14}H_{28}N_2O_6$: C, 52.48; H, 8.81, N, 8.74. Found: C, 52.07; H, 8.79; N, 8.65.

Example 3

[0027] Synthesis of dibutyl galactaramide. Diethyl galactarate (2.00 g, 7.5 mmol) was added to a solution of butylamine (1.40 g, 18.8 mmol) in EtOH (30 ml). After 20 hours stirring, the precipitate was filtered off and crystallized from DMSO/H_2O (yield 0.30 g, 0.9 mmol, 13%). ^{1}H-NMR (d_6-DMSO, 300 MHz, ppm): δ 0.88 (t, 6H), 1.29 (m, 4H), 1.40 (m, 4H), 3.11 (m, 4H), 3.78 (s, 2H, H_3, H_4), 4.11 (s, 2H, H_2, H_5), 4.39 (bs, 2H, OH_3, OH_4), 5.23 (bs, 2H, OH_2, OH_5), 7.55 (t, 2H, NH_1, NH_6). ^{13}C-NMR (d_6-DMSO, 300 MHz, ppm): 14.67, 20.41, 32.32, 38.86, 71.57, 174.07. Anal Calculated for $C_{14}H_{28}N_2O_6$: C, 52.48; H, 8.81, N, 8.74. Found: C, 51.58; H, 8.88; N, 8.50.

Example 4

[0028] Synthesis of dicyclohexyl D-ribaramide. D-Ribaric acid (lacton) (0.32 g, 2.0 mmol) was added to a solution of NEt_3 (0.25 ml) and cyclohexylamine (0.45 g, 4.5 mmol) in EtOH (20 ml). After 20 hours stirring, the solution was cooled to T = 4° C and filtered. Yield 0.14 g (0.41 mmol, 20%). ^{1}H-NMR (d_6-DMSO, 500 MHz, T = 100° C, ppm): δ 1.27 (m, 10H), 1.70 (m, 10H), 3.59 (bs, HDO + 1H), 3.97 (s, 2H), 7.52 (d, 1H, NH_6). ^{13}C-NMR (d_6-DMSO, 300 MHz, ppm): 25.90, 26.38, 33.42, 48.59, 73.24, 75.98, 173.12. Anal Calculated for $C_{17}H_{30}N_2O_5$: C, 59.63; H, 8.83, N, 8.18. Found: C, 59.30; H, 9.05; N, 8.03.

Example 5

[0029] Synthesis of dicyclohexyl D-glucaramide. D-Glucaro 6, 3 lactone (1.07 g, 5.6 mmol) was added to a solution of p-toluene sulfonic acid (0.042 g, 0.22 mmol) in EtOH (20 ml). At T = 50°C cyclohexylamine (1.10 g, 11.1 mmol) is dropped slowly to the solution. After one hour stirring, the solution was cooled to RT and H_2O (20 ml) was added. Evaporation till 10-15 ml gave a white precipitate which was filtered off and crystallized from EtOH (yield 0.89 g, 2.1 mmol, 38%). ^{1}H-NMR (d_6-DMSO, 300 MHz, ppm): δ 1.26 (m, 10H), 1.69 (m, 10H), 3.56 (bs, 2H), 3.68 (bs, 1H), 3.89 (bs, 2H), 3.96 (s, 1H), 4.61 (d, 1H), 4.69 (d, 1H), 5.36 (d, 1H), 5.45 (d,

1H), 7.31 (d, 1H, NH_1), 7.58 (d, 1H, NH_6). ^{13}C-NMR (d6-DMSO, 300 MHz, ppm): 25.57, 26.07, 33.16, 48.10, 48.25, 71.34, 72.57, 73.85, 74.04, 172.17, 172.98. Anal Calculated for $C_{18}H_{32}N_2O_6$: C, 58.05; H, 8.66, N, 7.52. Found: C, 58.11; H, 8.76; N, 7.46.

Example 6

[0030] Synthesis of dicyclohexyl D-mannaramide. D-Mannaric acid dilactone (0.45 g, 2.3 mmol) was added to a solution of cyclohexylamine (0.58 g, 5.9 mmol) in EtOH (20 ml). After 20 hours stirring, the solution was refluxed for two hours and after cooling the precipitate was filtered off and crystallized from EtOH (yield 0.05 g, 0.13 mmol, 6%). ^{1}H-NMR (d_6-DMSO, 300 MHz, ppm): δ 1 .24 (m, 10H), 1.70 (m, 10H), 3.59 (bs, 2H), 3.69 (t, 2H), 3.86 (t, 2H), 4.72 (d, 2H), 5.34 (d, 2H), 7.62 (d, 2H, NH). ^{13}C-NMR (d_6-DMSO, 300 MHz, ppm): 25.52, 26.10, 33.16, 48.26, 71.96, 72.27, 173.36. Anal Calculated for $C_{14}H_{28}N_2O_6$: C, 58.05; H, 8.66, N, 7.52. Found: C, 57.80; H, 8.74; N, 7.37.

Example 7

[0031] Synthesis of dicyclohexyl galactaramide. Diethyl galacterate (2.66 g, 10.0 mmol) was added to a solution of cyclohexylamine (2.68 g, 27.0 mmol) in EtOH (50 ml). After 20 hours stirring, the suspension was refluxed for three hours and after cooling the precipitate was filtered off, washed with H_2O/aceton 9:1 (3 x 25 ml) and H_2O (50 ml) and crystallized from DMSO (yield 0.84 g, 2.3 mmol, 23%). ^{1}H-NMR (d_6-DMSO, 300 MHz, ppm): δ 1.24 (m, 10H), 1.71 (m, 10H), 3.60 (bs, 2H), 3.76 (d, 2H), 4.09 (d, 2H), 4.38 (d, 2H), 5.18 (d, 2H), 7.26 (d, 2H, NH). ^{13}C-NMR (d_6-DMSO, 300 MHz, ppm): 25.08, 25.82, 32.95, 47.87, 71.39, 71.60, 172.74. Anal Calculated for $C_{14}H_{28}N_2O_6$: C, 58.05; H, 8.66, N, 7.52. Found: C, 57.88; H, 8.74; N, 7.42.

Example 8

[0032] Synthesis of dioctyl D-glucaramide. D-Glucaric acid (lactone) (1.35 g, about 7.0 mmol) was added to a solution of octylamine (1.85 g, 14.0 mol) in EtOH (30 ml). After 20 hours stirring, the suspension was refluxed for three hours and after cooling the precipitate was filtered off and. recrystallized twice from EtOH (yield 0.57 g, 1.3 mmol, 19%). ^{1}H-NMR (d_6-DMSO, 500 MHz, COSY, T = 50°C, ppm): δ 0.83 (t, 6H, 1.22 (m, 20 H), 1.37 (m, 4H), 3.04

(m, 4H), 3.65 (m, 1H, H_4), 3.82 (m, 1H, H_3), 3.88 (m, 1H, H_5), 3.94 (m, 1H, H_2), 4.56 (d, 1H, J = 6.9 Hz, OH_3), 4.70 (d, 1H, J = 4.7 Hz, OH_4), 5.31 (d, 1H, J = 5.2 Hz, OH_2), 5.48 (d, 1H, J = 6.3 Hz, OH_5), 7.56 (t, 1H, J = 5.9 Hz, NH_1), 7.80 (t, 1H, J = 5.9 Hz, NF_6). ^{1}H-NMR (d_6-DMSO, 500 MHz, COSY, 1 drop D_2O added, T = 50°C ppm): δ 0.83 (t, 6H), 1.22 (m, 20H), 1.37 (m, 4H), 3.04 (m, 4H), 3.65 (dd, 1H, $J_{4,5}$ = 6.2 Hz, $J_{3,4}$ = 3.5 Hz, H_4), 3.82 (t, 1H, $J_{2,3}$ = 3.7 Hz, H_3), 3.88 (d, 1H, H_5), 3.94 (d, 1H, H_2), ^{13}C-NMR (d_6-DMSO, 300 MHz, HMQC, T = 50°C, ppm): 14.65, 22.80, 27.10, 29.47, 29.72, 29.82, 31.98, 38.97, 71.08, 72.37, 73.55, 73.66, 172.70, 173.60. Anal Calculated for $C_{22}H_{44}N_2O_6$: C, 61.08; H, 10.25, N, 6.48. Found: C, 60.94; H, 10.41; N, 6.42.

Example 9

[0033] Synthesis of dioctyl D-mannaramide. D-Mannaric acid dilactone (2.14 g, 12.3 mmol) is added to a solution of octylamine (3.10 g, 5.9 mmol) in EtOH (50 ml). After 20 hours stirring, the suspension was refluxed for one hour and after cooling the precipitate was filtered off and crystallized from EtOH (yield 1.59 g, 3.7 mmol, 30%). ^{1}H-NMR (d_6-DMSO, 300 MHz, ppm): δ 0.87 (t, 6H), 1.25 (m, 20H), 1.42 (m, 4H), 3.08 (m, 4H), 3.70 (t, 2H), 3.88 (t, 2H), 4.79 (d, 2H), 5.41 (d, 2H), 7.87 (d, 2H, NH). ^{13}C-NMR (d_6-DMSO, 300 MHz, ppm): 14.91, 23.05, 27.31, 29.62, 29.99, 32.23, 39.28, 72.05, 72.38, 174.3. Anal Calculated for $C_{22}H44N_2O_6$: C, 61.08; H, 10.25, N, 6.48. Found: C, 60.84; H, 10.39; N, 6.40.

Example 10

[0034] Synthesis of dioctyl galactaramide. Diethyl galacterate (2.66 g, 10.0 mmol) was added to a solution of octylamine (2.64 g, 20.5 mmol) in EtOH (50 ml). After 20 hours stirring, the precipitate was filtered off and crystallized from DMSO (yield 3.00 g, 6.9 mmol, 69%). ^{1}H-NMR (d_6-DMSO, 300 MHz, ppm): δ 0.90 (bs, 6H), 1.30 (bs, 20H), 1.48 (bs, 4H), 3.14 (bs, 4H), 3.82 (bs, 2H), 4.08 (bs, 2H), 4.17 (bs, 2H), 4.85(bs, 2H), 7.33 (bs, 2H, NH). ^{13}C-NMR (d_6-DMSO, 300 MHz, ppm): 14.11, 22.37, 26.82, 28.95, 29.11, 29.61, 31.60, 38.97, 71.45, 173.34. Anal Calculated for $C_{22}H_{44}N_2O_6$: C, 61.08; H, 10.25, N, 6.48. Found: C, 61.15; H, 10.47; N, 6.44.

Example 11

[0035] Synthesis of dicitronellyl D-glucaramide. D-Glucaric acid (lactone) (2.90 g, about 14.0 mmol) was added to a solution of citronellylamine (5.00 g, 32.4 mmol) in EtOH (40 ml). After 20 hours stirring, the suspension was refluxed for three hours and after cooling, the precipitate was filtered off and recrystallized from 2-PrOH. Yield 2.30 g (4.8 mmol, 33%). ^{1}H-NMR (d_6-DMSO, 500 MHz, ppm): δ 0.86 (d, 6H), 1.09-1.45 (m, 10H), 1.58 (s, 6H), 1.66 (s, 6H), 1.95 (m, 4H), 3.12 (m, 4H), 3.71 (bs, 1H), 3.89 (bs, 1H), 3.92 (bs, 1H), 3.98 (bs, 1H), 4.63 (bs, 1H, 4.77 (bs, 1H), 5.10 (t, 2H), 5.35 (bs, 1H), 5.55 (bs, 1H), 7.57 (t, 1H), 7.84 (t, 1H). ^{13}C-NMR (d_6-DMSO, 300 MHz, ppm): 18.45, 20.15, 25.84, 26.44, 30.57, 36.99, 37.09, 37.25, 37.32, 37.53, 71.32, 72.47, 73.89, 74.18, 125.60, 131.37, 172.92, 173.97. Anal Calculated for $C_{26}H_{48}N_2O_6$: C, 64.43; H, 9.98, N, 5.78. Found: C, 64.13; H, 10.02; N, 5.75.

Example 12

[0036] Synthesis of didodecyl D-glucaramide. D. Glucaric acid (lactone) (0.81 g, about 3.9 mmol) was added to a solution of dodecylamine (1.94 g, 10.5 mmol) in EtOH (25 ml). After 72 hours stirring, the suspension was refluxed for three hours and after cooling, the precipitate was filtered off and recrystallized from DMSO. Yield 1.30 g (2.4 mmol, 61%). ^{1}H-NMR (d_6-DMSO, 300 MHz, T = 100° C, ppm.): δ 0.89 (t, 6H), 1.29 (m, 36H), 1.47 (t, 4H), 3.13 (m, 4H), 3.75 (m, 1H), 3.92 (m, 1H), 3.97 (m, 1H), 3.99 (m, 1H), 4.50 (bs, 2H), 5.05 (bs, 2H), 7.30 (t, 1H), 7.53 (t, 1H). ^{13}C-NMR (d_6-DMSO, 300 MHz, T = 100° C, ppm): 14.07, 22.34, 26.79, 28.99, 29.12, 29.33, 31.63, 38.86, 71.15, 72.41, 73.37, 73.55, 172.37, 173.41. Anal Calculated for $C_{30}H_{60}N_2O_6$: C, 66.14; H, 11.10, N, 5.14. Found: C, 66.14; H, 11.05; N, 5.12.

Example 13

[0037] Synthesis of didodecyl D-mannaramide. D-Mannaric acid dilactone (0.43 g, 2.5 mmol) was added to a solution of dodecylamine (1.12 g, 6.1 mmol) in EtOH (20 ml). After 72 hours stirring, the precipitate was filtered off and crystallized from DMSO (yield 0.35 g, 3.7 mmol, 26%). ^{1}H-NMR (d_6-DMSO, 300 MHz, T = 100° C, ppm): δ 0.89 (t, 6H), 1.29 (m, 36H), 1.47 (t, 4H), 3.14 (m, 4H), 3.77 (d, 2H), 3.95 (d, 2H), 4.61 (bs, 2H), 5.08 (bs, 2H), 7.54 (t, 1H). ^{13}C-NMR (d_6-DMSO, 300 MHz, T = 100° C, ppm): 14.05, 22.35, 26.77, 28.98, 29.11, 29.34,

31.63, 38.96, 71.96, 72.31, 173.60. Anal Calculated for $C_{30}H_{60}N_2O_6$: C, 66.14; H, 11.10, N, 5.14. Found: C, 65.76; H, 11.01; N, 5.11.

Example 14

[0038] Synthesis of didodecyl galactaramide. Diethyl galacterate (2.66 g, 10.0 mmol) was added to a solution of dodecylamine (3.75 g, 20.5 mmol) in EtOH (50 ml). After 72 hours stirring, the precipitate was filtered (yield 4.87 g, 8.9 mmol, 89%). Owing to the low solubility in several solvents tested, no proper NMR spectra could be obtained.

Example 15

[0039] Synthesis of dicyclododecyl D-glucaramide. D-Glucaro 6, 3-lacton (7.67 g, 40.0 mmol) was added to a solution of cyclododecylamine (14.93 g, 81.6 mmol) in 2-methoxyethanol (125 ml). The reaction mixture was heated slowly till T = 120° C in three hours and kept at this T for four hours. After cooling, the precipitate was filtered off and recrystallized from DMSO and EtOH. Yield 9.50 g (17.6 mmol, 44%). ^{1}H-NMR (d_6-DMSO, 300 MHz, T = 100° C, ppm): δ 1.36 (m, 36H), 1.60 (m, 4H), 3.75 (m, 1H), 3.97 (m, 5H), 4.47 (bs, 2H), 6.98 (bs, 1H), 7.21 (bs, 1H). ^{13}C-NMR (d_6-DMSO, 300 MHz, T = 100° C, ppm): 22.03, 23.97, 24.14, 30.59, 45.45, 71.21, 72.32, 73.33, 73.71, 171.76, 172.76. Anal Calculated for $C_{30}H_{56}N_2O_6$: C, 66.63; H, 10.44, N, 5.18. Found: C, 67.00; H, 11.30; N, 4.97.

Example 16

[0040] Synthesis of dicyclododecyl D-mannaramide. D-Mannaric acid. dilactone (1.99 g, 11.4 mmol) was added to a solution of cyclododecylamine (0.58 g, 5.9 mmol) in EtOH (20 ml). After 20 hours stirring, the solution was refluxed for two hours and after cooling, the precipitate was filtered off and crystallized from EtOH and DMSO (yield 0.51 g, 0.94 mmol, 10%). ^{1}H-NMR (d_6-DMSO, 300 MHz, T = 100° C, ppm): δ 1.36 (m, 36H), 1.61 (m, 8H), 3.75 (bs, 2H), 3.93 (bs, 4H), 4.58 (bs, 2H), 5.07 (bs, 2H), 7.24 (d, 2H, NH). ^{13}C-NMR (d_6-DMSO, 300 MHz, T = 100° C, ppm): 22.05, 23.99, 24.17, 30.62, 45.68, 72.23, 173.01. Anal Calculated for $C_{30}H_{56}N_2O_6.0.25\ C_2H_6SO$: C, 65.01; H, 10.46, N, 5.05. Found: C, 65.08; H, 10.29; N, 5.05.

Example 17

[0041] Synthesis of dicyclododecyl galactaramide. Diethyl galacterate (2.67 15 g, 10.0 mmol) was added to a solution of cyclododecylamine (3.78 g, 20.7 mmol) in EtOH (50 ml). After 48 hours stirring, the precipitate was filtered and washed with H_2O and EtOH (yield 2.43 g, 4.5 mmol, 45%). Owing to the low solubility in several solvents tested, no proper NMR spectra could be obtained.

Example 18

[0042] Synthesis of di-8-pentadecyl D-glucaramide. D-Glucaric acid (lactone) (2.53 g, about 12.7 mmol) is added to a solution of 8-aminopentadecane (5.17 g, 26.1 mmol) in EtOH (35 ml). After 20 hours stirring, the suspension was refluxed for 20 hours and recrystallized from EtOH/H_2O (3x). Yield 0.72 g (1.3 mmol, 10%). ^{1}H-NMR (d_6-DMSO, 300 MHz, ppm): δ 0.86 (t, 12H), 1.23 (m, 32H), 1.36 (t, 8H), 3.69 (bs, 3H), 3.88 (bs, 1H), 3.94 (bs, 1H), 3.99 (m, 1H), 4.56 (bs, 1H), 4.71 (bs, 1H), 5.33 (bs, 1H), 5.47 (bs, 1H), 7.15 (d, 1H), 7.41 (d, 1H). ^{13}C-NMR (d_6-DMSO, 300 MHz, T = 100° C, ppm): 14.86, 23.03, 26.37, 29.58, 29.68, 32.21, 35.36, 48.78, 48.97, 71.43, 71.73, 73.85, 74.30, 172.52, 173.71. Anal Calculated for $C_{30}H_{60}N_2O_6$: C, 67.09; H, 11.26, N, 4.89. Found: C, 66.98; H, 11.38; N, 4.90.

Example 19

[0043] Synthesis of dioleyl D-glucaramide. D-Glucaric acid (lactone) (1.27 g, about 6.5 mmol) was added to a solution of oleylamine (3.87 g, 14.4 mmol) in EtOH (30 ml). After 20 hours stirring, the suspension was refluxed for 0.5 hour and recrystallized from EtOH (2x) and DMSO. Yield 0.72 g (1.4 mmol, 21%). ^{1}H-NMR (d_6-DMSO, 300 MHz, ppm): δ 0.88 (t, 6H), 1.28 (in, 44H), 1.45 (m, 4H), 2.01 (m, 8H), 3.07 (bs, HDO + 4H), 3.75 (m, 1H), 3.91 (m, 1H), 3.96 (m, 1H), 3.98 (m, 1H), 4.38 (bs, 1H), 4.52 (bs, 1H), 5.10 (bs, 2H), 5.35 (m, 4H), 7.32 (bs, 1H), 7.55 (bs, 1H). ^{13}C-NMR (d_6-DMSO, 500 MHz, T = 100° C, ppm): 14.36, 22.61, 27.04, 27.30, 27.34, 29.24, 28.28, 29.38, 29.44, 29.48, 29.62, 29.68, 29.76, 29.80, 31.88, 39.12, 39.20, 71.40, 72.67, 73.67, 73.86, 130.3, 172.68, 173.68. Anal Calculated for $C_{42}H_{80}N_2O_6$: C, 71.14; H, 11.37, N, 3.95. Found: C, 70.87; H, 11.43; N, 3.97.

Example 20

[0044] Synthesis of 3-*O*-methyl-dicyclohexyl D-glucaramide. 3-*O*-Methyl diethyl D-glucarate (0.54 g, 1.9 mmol) was added to a solution of cyclohexylamine (0.48 g, 4.8 mmol) in EtOH (20 ml). After 20 hours stirring, the precipitate was filtered off. Yield 0.20 g (0.75 mmol, 39%). ^{1}H-NMR (d_6-DMSO, 300 MHz, ppm): δ 1.26 (m, 10H), 1.70 (m, 10H), 3.32 (s, 3H), 3.59 (m, 2H), 3.65(m, 1H), 3.74 (m, 1H), 3.89 (m, 1H), 4.08 (m, 1H), 4.78 (m, 1H), 5.45 (bs, 2H), 7.40 (d, 1H), 7.51 (d, 1H). ^{13}C-NMR (d_6-DMSO, 300 MHz, ppm): 24.53, 26.13, 33.21, 48.20, 60.95, 72.35, 73.24, 74.51, 81.85, 172.00, 172.50. Anal Calculated for $C_{19}H_{34}N_2O_6$: C, 59.05; H, 8.87, N, 7.25. Found: C, 58.91; H, 8.90; N, 7.27.

Example 21

[0045] Synthesis of 3-*O*-methyl-didodecyl D-glucaramide. 3-O-Methyl diethyl D-glucarate (0.60 g, 2.1 mmol) was added to a solution of cyclododecylamine (0.93 g, 5.0 mmol) in EtOH (20 ml). After 72 hours stirring, the precipitate was filtered off and crystallized from EtOH. Yield 0.55 g (0.98 mmol, 46%). ^{1}H-NMR (d_6-DMSO, 300 MHz, T = 100° C, ppm): δ 0.84 (t, 6H), 1.26 (m, 36H), 1.45 (m, 4H), 3.34 (s, 3H), 3.70 (m, 1H), 3.80 (m, 1H), 3.95 (m, 1H), 4.09 (m, 1H), 4.58 (bs, 1H), 5.03 (bs, 2H), 7.32 (bs, 1H), 7.42 (bs, 1H). ^{13}C-NMR (d_6-DMSO, 300 MHz, T = 100° C, ppm): 14.08, 22.38, 26.83, 29.03, 29.16, 28.38, 31.66, 38.97, 60.02, 72.06, 72.72, 73.91, 81.39, 172.37, 173.15. Anal Calculated for $C_{31}H_{62}N_2O_6$: C, 66.63; H, 11.18, N, 5.01. Found: C, 66.63; H, 11.33; N, 5.04.

Example 22

[0046] Synthesis of 3-*O*-methyl-dicyclododecyl D-glucaramide. 3-*O*-Methyl diethyl D-glucarate (0.60 g, 2.1 mmol) is added to a solution of cyclododecylamine (0.98 g, 5.3 mmol) in EtOH (20 ml). After 20 hours stirring, the precipitate was filtered off and crystallized from EtOH. Yield 0.30 g (0.54 mmol, 25%). ^{1}H-NMR (d_6-DMSO, 300 MHz, T = 100° C, ppm): δ 1.33 (m, 36H), 1.57 (m, 8H), 3.35 (s, 3H), 3.69 (bs, 1H), 3.77 (bs, 1H), 3.93 (bs, 3H), 4.09 (bs, 1H), 4.47 (bs, 1H), 5.03 (bs, 2H), 7.02 (d, 1H), 7.12 (d, 1H). ^{13}C-NMR (d_6-DMSO, 300 MHz, T = 100° C, ppm): 22.02, 23.97, 24.16, 30.61, 45.52, 60.06, 71.91, 72.62, 72.76, 73.86, 81.43,

171.71, 172.42. Anal Calculated for $C_{31}H_{58}N_2O_6$: C, 67.11; H, 10.54, N, 5.05. Found: C, 65.64; H, 10.44; N, 5.00.

Example 23

[0047] Synthesis of N_1-cyclododecyl, N_6-cyclohexyl D-glucaramide. Cyclohexylammonium 6-(N-cyclohexyl)-D-glucaramide-1-ate (0.60 g, 2.1 mmol) was added to a solution of Dowex H^+ (1 x 8) in H_2O (40 ml). After 30 minutes stirring the suspension is filtered and washed thoroughly with H_2O. The filtrate was evaporated and the crude 6-(N-cyclohexyl)-D-glucaramide (lacton) was added to a solution of p-toluene sulfonic acid (0.038 g, 0.20 mmol) in EtOH (20 ml). At T = 50°C cyclododecylamine (0.47 g, 2.6 mmol) was dropped slowly to the solution. After one hour stirring the solution was cooled to T = 4°C and recrystallized from DMSO/H_2O. Yield 0.13 g (0.33 mmol, 13%). ^{1}H-NMR (d_6-DMSO, 300 MHz, T = 100° C, ppm): 1.13-1.75 (m, 32 H), 3.59 (bs, 1H), 3.70 (m, 1H), 3.92 (m, 1H), 3.94 (m, 2H), 3.99 (m, 1H), 4.49 (bs, 1H), 4.63 (bs, 1H), 5.20 (bs, 1H), 5.33 (bs, 1H), 7.13 (bs, 1H), 7.45 (bs, 1H). ^{13}C-NMR (d_6-DMSO, 300 MHz, T = 95° C, ppm): 22.20, 22.27, 24.20, 24.38, 25.05, 25.89, 30.86, 32.80, 32.84, 45.65, 48.15, 71.41, 72.68, 73.65, 73.90, 172.05, 172.80.

Example 24

[0048] Solvent scope of N,N′-dialkylaldaramides (1%) or N,N′-dialkylpentaramides (1%) 1-24 refers to the compounds prepared in Examples 1-23)

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23
Hexadecane	ns	Ns	ns	ns	ns	-	ns	ns	ns	ns	p	ns	ns	ns	ns	ns	ns	s	p	ns	p	ns	-
Cyclohexane	ns	Ns	ns	ns	G	ns	ns	ns	ns	ns	g*	ns	ns	ns	g	ns	ns	s	p	ns	g	g	p
p-xylene	ns	Ns	ns	p	G	-	ns	ns	ns	ns	p	ns	ns	ns	g	ns	ns	s	p	p	p	p	p
Toluene	ns	Ns	ns	p	G	ns	ns	ns	ns	ns	p	ns	ns	ns	g	ns	ns	s	p	p	p	p	-
n-butylacetate	ns	Ns	ns	p	G	ns	ns	ns	ns	ns	p	ns	ns	ns	g	ns	ns	s	c	p	c	p	p
1,2-dichloroethane	ns	Ns	ns	s	cg	p	ns	ns	ns	ns	s	p	c	ns	g	c	ns	s	c	c	p	p	-
2-octanol	P	Ns	ns	p	cg	-	ns	ns	ns	ns	s	ns	p	ns	s	s	ns	s	c	p	c	p	-
2-propanol	C	C	c	p	cg	-	c	c	c	ns	s	ns	c	ns	c	cg	ns	s	c	c	p	p	-
Ethanol	C	C	c	s	cg	-	c	c	c	c	s	p	ns	ns	cg	p	ns	s	c	c	p	p	-
Dimethylsulfoxide	S	C	s	s	cg	-	c	s	c	s	s	s	c	ns	cg	p	ns	s	c	s	p	p	-
Water	ns	Ns	ns	ns	ns	-	ns	ns	ns	ns	c	ns	ns	ns	ns	ns	ns	p	ns	ns	ns		-
silicon oil	-	-	-	--	G	-	-	-	-	-	-	-	-	-	g	-	ns	s	-	-	v	-	v
methyl laurate	-	-	-	-	G	-	-	-	-	-	-	-	-	-	g	-	ns	s	-	-	-	-	p
methyl benzoic acid	-	-	-	-	S	-	-	-	-	-	-	-	-	-	g	-	ns	s	-	-	p	-	p
2-methoxyethanol	-	-	-	-	S	-	-	-	-	-	-	-	-	-	c	-	ns	s	-	-	-	-	p

g = gelation, s = soluble, p = precipitates, c = crystallizes, ns = not soluble, v = viscous, g* = unstable gel, precipitates, cg = crystalline gel

Example 25

[0049] Gelation of N,N′-dialkylaldaramides (1%) in mixtures of solvents

	11 (cit-Glu-cit)	12 (12-Glu-12)	15 (C12-Glu-C12)
cyclohexane	g*	ns	**g**
cyclohexane/dioxane 1:1	s	-	cg
Dioxane	s	c	cg
dioxane/H_2O 3:1	s	c	cg
dioxane/H_2O 2:1	s	c	cg
dioxane/H_2O 1:1	p	ns	**g**
dioxane/H_2O 1:2	p	ns	ns
H_2O	c	ns	ns

g = gelation, s = soluble, p = precipitates, c = crystallizes, ns = not soluble, g* = unstable gel, precipitates , cg = crystalline gel

Example 26

[0050] Addition of a solution of the gelling agent (10% in NMP, 0.05 ml) to an organic solution (0.5 ml, "cold gelation")

	5 (C6-Glu-C6)	12 (12-Glu-12)	15 (C12-Glu-C12)	18 (B13-Glu-B13)
cyclohexane	p	p	g	s
methyl laurate	c	p	g	s
Toluene	c	p	g	s
n-butyl acetate	c	p	g	s
1,2-dichloroethane	c	p	c	s
silicon oil	-	-	g	-
Acetone	-	-	cg	-
benzaldehyde	-	-	s	-
Chloroform	-	-	s	-
diethyl ether	-	-	g	-
ethylacetate	-	-	p	-
Heptane	-	-	p	-
Hexane	-	-	p	-
Acetonitril	-	-	g	-
tetrahydrofuran	-	-	c	-

Example 27

[0051] Maximum gelator concentration of 5 (C6-Glu-C6) and 15 (C12-Glu-C12)

	5 (in %) (C6-Glu-C6)	15 (in %) (C12-Glu-C12)
cyclohexane	< 5	< 5
methyl laurate	< 2.5	< 5
silicon oil (Dow Corning 702)	< 2.5	< 50
Toluene	< 2.5	< 50
n-butylacetate	< 2.5	< 50
1,2-dichloroethane	< 5	< 50

Example 28

[0052] Phase diagram of 5 (C6-Glu-C6) and 15 (C12-Glu-C12))dropping ball method)

[0053] The phase diagram of 5 (C6-Glu-C6) and 15 (C12-Glu-C12) was determined (see Figure 1) by using the dropping ball method (A. Takashi, M. Sakai, T. Kato, *Polym. J.*, 12 (1980) 335-341, F.S. Schoonbeek, J.H. van Esch, R. Hulst, R.M. Kellogg, B.L. Feringa, *Chem. Eur. J.*, 6 (2000) 2633-2643). A linear correlation was observed between the Tm^{-1} and the logarithm of the mole fraction of 15 (C12-Glu.-C12) in cyclohexane, silicon oil and p-xylene, as expected for the dissolution process of crystals (gels) (K. Murata, M. Aoki, T. Suzuki, T. Harada, H. Kawabata, T. Komori, F. Ohseto, S. Shinkai, *J. Am. Chem. Soc.*, 116 (1994) 6664-6676).